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Insight on the development of molybdenum oxide-based photocatalyst towards pharmaceutical pollutants abatement

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Abstract. The presence of pharmaceutical compounds in the environment has given an unparallel impact on the human and environment. Till the present day, several methods have been imposed for the pharmaceutical pollutants' removal. However, these methods suffer from some drawbacks which limited their application in wastewater treatment. As a new advanced technology, the photocatalysis method has been considered as an efficient method to eliminate the pharmaceutical pollutants in the water matrix. Molybdenum oxide (MoO₃) photocatalyst has captivated global interest due to its non-toxicity, good thermal and chemical stability, and remarkable optical properties. However, the limitations of MoO₃ material have hindered its theoretical performance in eliminating distinct pharmaceutical pollutants. Some modification strategies have been proposed for MoO₃ photocatalyst which results in the development of several modified MoO₃ photocatalysts towards a broad range of pharmaceutical pollutants removal.

1. Introduction

In this modern human society, the advent of the industrial revolution and urbanization processes have contributed significantly to the increasing of pharmaceutical compounds in the water matrix. The presence of pharmaceutical compounds in the water matrix not only demonstrated an adverse effect on human health but also results in an unparallel impact on the sustainability of ecological balance. To date, the increase in the production of pharmaceutical products can be ascribed to a few factors including change of lifestyle, increasing of the human population, climate change, and the evolution of new species of bacteria [1]. Explicitly, the pharmaceutical industry produces a variety of products every year which comprises cosmetic, antibiotic, analgesic, tranquillizers and other medical products. However, due to direct discharge of treated effluent, abusive uses of pharmaceutical products, and improper disposal of expired pharmaceutical products, large distribution of pharmaceutical pollutants are detected in the environment especially in the aquatic domain [2]. Thus, significant efforts have been made to reduce and destroy the pharmaceutical compounds in the aquatic domain.

To date, many treatment methods are available for pharmaceutical pollutants abatements such as coagulation-flocculation, adsorption, membrane separation, anaerobic digestion, and ion-exchanges [3]. These methods, however constraint by some drawbacks such as high cost of operation and maintenance, risk generation of secondary pollutants, unable to degrade or remove the low concentration of pollutant



and production of sludge which significantly hindered the high performance and efficiency in removing pharmaceutical pollutants from wastewater [4]. Therefore, current research is an exertion to develop efficient and environmentally-friendly technology capable to remove water pollutants especially pharmaceutical products from the water matrix. Over the years, the photocatalysis approach has gained tremendous interest for wastewater treatment due to its high efficiency, low cost, mild operation condition, and green technology [5].

The Photocatalysis approach is an advanced technology capable to destroy or eliminate pollutants in the water event at a low concentration. Photocatalysis approach is known as a promising method to remove the pollutants from wastewater due to its environmentally-friendliness, simple operation, low cost of operation, high reusability and high efficiency in removing water pollutants [6]. Furthermore, this method is based on the principle of the utilization of the ultimate oxidants, hydroxyl radical ($\bullet\text{OH}$). Figure 1 illustrated the general principle of the photocatalysis method. Explicitly, the photocatalysis method works in the presence of light illumination. When the photocatalyst is exposed to light irradiation, the electron at the valence band will excite to the conduction band of photocatalyst and leaving electron vacancy known as hole (h^+). The excited electrons are then well distributed to the conduction band of photocatalyst and further utilized for the reduction of oxygen molecule (O_2) to superoxide anion radical ($\bullet\text{O}_2^-$). Meanwhile, photogenerated holes is then undergoes oxidation process with water molecule (H_2O) to form the ultimate oxidant, hydroxyl radical. Both superoxide anion and hydroxyl radicals are among the main reactive oxygen species which play significant roles in degrading water pollutants in the hydrosphere.

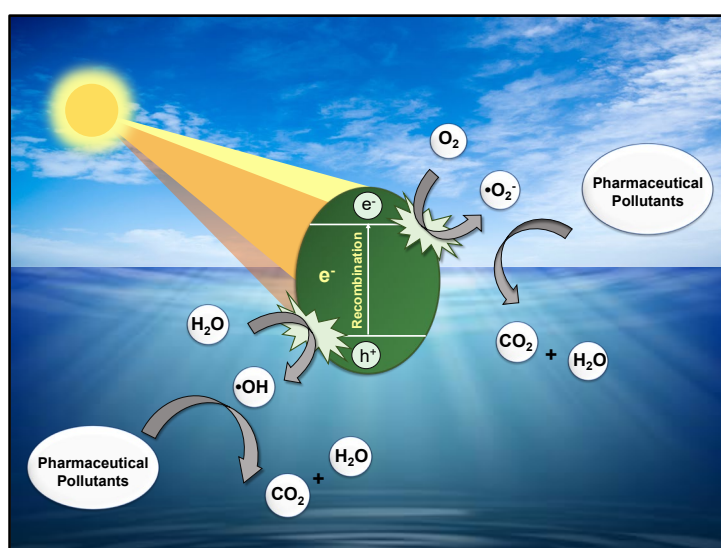


Figure 1. Principle of conventional photocatalytic degradation [1].

The selection of photocatalyst material is one of the critical challenges that significantly affect the photocatalytic performance towards removing of water pollutants. Till the present day, numerous metal oxide semiconductor materials such as TiO_2 [7], ZnO [8], Fe_2O_3 [9], and CuO [10] have been extensively studied for the degradation of pharmaceutical pollutants due to their unique characteristics such as good absorption of light energy, high charge conductivity, non-toxic material, and high photocatalyst stability. These photocatalysts, however, are limited by some shortcomings such as poor visible light response range, and high recombination of photogenerated charges which restricted their application for real wastewater application. On the other hand, MoO_3 is a widely known catalyst material that has been utilized in many applications including lithium-ion batteries, gas sensors, solar cells and supercapacitors due to its remarkable its physical and optical characteristics. Interestingly, recently, molybdenum oxide (MoO_3) has aroused as a promising material for wastewater treatment due to its astonishing properties

such as adequate visible light response range, anisotropic crystal growth and controllable morphology and high chemical stability [11].

MoO₃ is a semiconductor material which predominantly exists in three polymorphs phases including α -MoO₃, β -MoO₃ and h-MoO₃. The bandgap of MoO₃ semiconductor typically lies in the range of 2.8 – 3.2 eV [12]. Although h-MoO₃ demonstrated wider bandgap energy (2.8 - 3.0 eV) [13] as compared to the other phases (2.69 - 2.76 eV) [14], yet, it exhibits more superior photocatalytic activity due to its unique characteristics especially the one dimensional (1-D) and tunnel structure which provides high electron transferability, high accessibility of active sites and high surface-to-volume ratio [13]. Nevertheless, the bottlenecks of MoO₃ photocatalyst such as rapid photogenerated charges recombination, low surface area and accessibility of active sites, and inadequate conduction band for superoxide anion radicals (O₂⁻) formation have hindered the high performance of MoO₃ photocatalyst towards degrading of pharmaceutical pollutants [15]. Hence, some modifications should be made to the MoO₃ photocatalyst in order to overcome these shortcomings.

Recently, several studies have reported on the development of modified MoO₃ photocatalysts toward a distinct range of pharmaceutical pollutants removal which comprise of few strategies such as introducing metal element [16], surface modifying the surface morphology [17] and utilizing the graphitic carbon nitride as support material [18,19]. Despite the numerous reported works on the development of modified MoO₃ photocatalyst for various pharmaceutical pollutants removal, yet, there is no available study has reviewed thoroughly the potential of these modified MoO₃ photocatalysts towards the pharmaceutical pollutants' removal. This work will thoroughly scrutinize the performance of modified MoO₃ photocatalyst towards various pharmaceutical pollutants removal as well as the mechanistic involved during the photocatalysis process.

2. Modification of Molybdenum Oxide Photocatalyst

The emerging of molybdenum oxide as a promising photocatalyst recently has brought new insight into the utilization of MoO₃ based photocatalyst for the water purification process. The remarkable physicochemical and optical properties are among the compared to the other metal oxide materials. Despite its potential in degrading the targeted water pollutants, the MoO₃ photocatalyst exhibits poor adequacy in harvesting visible light energy and rapid recombination of the excitons. In turn, results in the low generation of photoexcited charges and photocatalytic activity. Besides, the position of band energy is not favorable for the formation of superoxide anion radicals which limited its performance towards the degradation of pharmaceutical pollutants. Attributed to the aforementioned problems, significant efforts have been made on the MoO₃ photocatalyst to tackle these drawbacks which involved the development of several modified MoO₃ photocatalysts. Notably, several modification strategies can be applied to the MoO₃ photocatalyst such as modifying surface morphology and synthesizing of heterojunction photocatalyst. However, to date, the development of heterojunction photocatalyst has gained tremendous interest in the research area. Table. 1 below summarized the development of modified MoO₃ based photocatalyst and its efficiency towards the degradation distinct range of pharmaceutical pollutants. Remarkably, the development of modified MoO₃ photocatalysts has shown great success toward the degradation of various pharmaceutical pollutants.

Table 1 Summary of modified MoO₃ photocatalyst for various pharmaceutical pollutants removal.

Photocatalyst	Pollutant's Concentration (mg L ⁻¹)	Light Source	Surface Area (m ² g ⁻¹)	Bandgap (eV)	pH	Catalyst Dosage (g L ⁻¹)	Pollutant's Degradation (%)	Ref.
MoO ₃ /Bi ₂ O ₃ /g-C ₃ N ₄	TC-HCl: 10	Solar light	-	2.32	6.0	0.6	98.0 (120 min)	[20]
AgBr/h-MoO ₃	TMP: 10	Solar light	6.66	2.35	-	0.1	100.0 (30 min)	[11]
Bi ₅ O ₇ I/MoO ₃	IBU: 15	Visible light	6.80	1.74	6.8	0.2	89.2 (120 min)	[15]
MoO ₃ /g-C ₃ N ₄	OFX: 10	Visible light	-	2.56	3.0	0.6	94.4 (120 min)	[18]
BiVO ₄ /MoO ₃	TC-HCl: 140	Solar light	8.57	2.86	4.5	0.5	99.8 (160 min)	[21]
ZnIn ₂ S ₄ @MoO ₃	TC-HCl: 30	Visible light	-	-	-	0.15	94.5 (90 min)	[22]
SiO ₂ /MoO ₃	MZ: 10	Visible light	202.00	2.58	5.0	0.3	67.4% (180 min)	[17]
MoO ₃ /Ag/g-C ₃ N ₄	OFLX: 20	Visible light	51.70	-	-	0.2	96% (100 min)	[16]
TMO	CIP: 16	UV light	-	3.26	10	1.0	90% (75 min)	[23]
BiMoO ₆	OFLX: 10	Solar light	-	2.27	6.0	1.0	71% (90 min)	[24]
Mo-PCN	TCH-HCl: 20	Visible light	50.43	2.65	-	0.2	61.7% (60 min)	[25]
MoO ₂	PMOL: 5	Visible light	30.40	2.50	-	0.5	71.0% (240 min)	[26]
5%MoO ₃ /Mg Al-LDH-C	MNZ: 10	Visible light	-	-	5.0	1.5	77.0% (160 min)	[27]

2.1. Graphitic Carbon Nitride (g-C₃N₄) as Support

Notably, MoO₃ photocatalyst is an n-type semiconductor that can transfer the electron to the other semiconductor. Despite, the high photocatalytic activity of MoO₃ photocatalyst, its performance towards the degradation of pharmaceutical pollutants is still beyond the theoretical expectation due to various factors which included the inability to form superoxide anion radical (one of the reactive oxygen species). Interestingly, the hybridization of MoO₃ with graphitic carbon nitride (g-C₃N₄) provides a significant improvement in the photocatalytic activity of the MoO₃ photocatalyst. The g-C₃N₄ not only enhances the formation of superoxide anion radical but also provides a large surface area for more accessibility of active sites.

Several developments of MoO₃@g-C₃N₄ photocatalysts have been done towards the degradation of various pharmaceutical pollutants recently. Chen et al. (2019) has efficaciously synthesized the modified MoO₃ photocatalyst by introducing the g-C₃N₄ as support. Remarkably, the synthesized photocatalyst show superior photocatalytic activity relative to the pristine MoO₃ and g-C₃N₄ photocatalyst with removal efficiency of 94.4% within 120 minutes [18]. The utilization of g-C₃N₄ as shows is reported to exhibit a large surface area for more accessible of active sites. Moreover, this heterostructure photocatalyst demonstrated excellent segregation of electron-hole which contributed significantly to the remarkable ofloxacin removal. The g-C₃N₄ also provides more absorption of light energy for efficient photocatalytic activity. Due to the lower position of the conduction band of MoO₃, the formation of superoxide anion radicals is hindered. Thus, the introduction of g-C₃N₄ which favors the formation of

superoxide anion radicals indirectly decrypt this issue. Moreover, the g-C₃N₄ significantly tuned the large bandgap of MoO₃ which provides high light absorption at the visible light region.

2.2. *Introducing of Metal Element*

The strategy of introducing metal elements onto the single or heterostructure photocatalyst surface is a common practice nowadays. Ahead of photocatalytic experiments, numerous works have unveiled the potential of metal elements in boosting the photocatalytic activity. In the eliminating of pharmaceutical pollutants, a recent study has reported on the synthesizing of MoO₃/Ag/g-C₃N₄ towards the degradation of ofloxacin [16]. Interestingly, in this study, the synthesized MoO₃/Ag/g-C₃N₄ exhibits superior photocatalytic performance under visible light illumination. Impressively, 96% of the ofloxacin pollutant is degraded within 100 minutes. The Ag nanoparticles (Ag NPs) provide a channel for electrons transfer from conduction band MoO₃ to the valence band of g-C₃N₄. Besides, the Ag NPs also acts as a recombination centre for the photogenerated electron-hole. Hence, results in highly segregation of charge carriers. Besides, the introduction of metal, particularly noble metals can significantly enhance the performance of photocatalytic activity due to the presence of localized surface plasmonic resonance (LSPR) effects which suppress the recombination of electrons and holes. In addition, the LSPR effect substantially enhanced the formation of superoxide anion radicals due to the presence of more electron species. This study evinced that the introduction of metal elements is a remarkable strategy to improve the photocatalytic activity of the MoO₃ photocatalyst.

2.3. *Incorporation of Homogeneous Photocatalyst into Heterogeneous Photocatalyst*

Modification of MoO₃ photocatalyst is an excellent strategy to improve the photocatalytic activity of MoO₃ photocatalyst. Typically, the modification strategy involved the introduction of metal elements or semiconductor material either as support or secondary photocatalyst material. Recently, several studies have reported on the development of tertiary photocatalyst by combining a single photocatalyst with binary photocatalyst. Impressively, this strategy yield highly photocatalytic performance compares to the binary photocatalyst and single photocatalyst. For instance, Alnaggar et al. (2021) reported the successful fabrication of MoO₃/Bi₂O₃/g-C₃N₄ (MBG) heterostructure towards the degradation of tetracycline hydrochloride (TCH) compound. Interestingly, this tertiary photocatalyst significantly suppressed the recombination of photoproducted electrons and holes. Besides, the synthesized MBG photocatalyst demonstrated significant tuned of the large bandgap of MoO₃ photocatalyst from 2.75 eV to 2.32 eV [20]. Thus, increase the light absorption in the visible light region which results in the improvement of efficiency in degrading tetracycline hydrochloride compound. The addition of peroxydisulfate (PMS) as a sulfate radical source into the photocatalytic system shows remarkable photocatalytic performance towards TCH removal. In this study, the MoO₃ with (Mo⁶⁺/Mo⁵⁺) redox cycle plays a substantial role in the activating of PMS to produce sulfate radicals. Impressively, the removal efficiency of TCH using MBG photocatalyst in the presence of PMS increases drastically from 64% to 98% within 120 minutes of solar light irradiation. This finding concluded that the addition of oxidation agents such as sulfate anion radical into the photocatalytic system can help to degrade more TCH compounds.

2.4. *Modifying Surface Morphology*

Among the unique characteristic of MoO₃ as photocatalyst material is its controllable morphology. MoO₃ predominantly exists as thermodynamically stable of α -MoO₃ state. The application of α -MoO₃ photocatalyst in wastewater treatment has gained significant interest due to its photocatalytic efficiency. However, it is limited by the low surface area which results in low accessibility of active sites for the photocatalytic reaction. Recent studies have focused on developing one dimensional (1D) photocatalyst in order to increase the surface area of the single photocatalyst. Cai et al. (2021) has reported on the fabrication of h-MoO₃ photocatalyst towards trimethoprim abatement. Compare to the other MoO₃ phases, h-MoO₃ exhibits dominant photocatalytic activity due to the anisotropic crystal growth, large surface to volume ratio, direct electrical transfer, high absorption of light energy, and low bandgap

energy. However, the performance of h-MoO₃ is limited by its high charge carrier's recombination. Interestingly, the incorporation of AgBr onto h-MoO₃ significantly improved the photocatalytic activity of h-MoO₃. The presence of Ag NPs which are produced by the reduction of Ag⁺ can act as an electron trap centre to suppress the recombination rate of photogenerated charges [11]. However, the escalating of Ag NPs deposition demonstrated the non-LSPR effect which reduces the absorption of light for the photocatalysis process.

3. Mechanism of Modified MoO₃ Photocatalyst

The developments of modified MoO₃ photocatalysts have imparted substantial information on the potential of altered MoO₃ photocatalyst for efficient photocatalytic enactment. In addition, these developments also unveil the discovery of new mechanistic photocatalytic reactions and the reactive species that play a substantial role in the substrate's removal. In the conventional photocatalytic mechanism, the heterojunction photocatalyst materials typically exhibit type II p-n junction interaction. However, recent studies have exposed the formation of the Z-scheme mechanism which has been authenticated by scavenger experiments and characterization studies. Compare to the conventional heterojunction photocatalytic mechanism, the inception of the Z-scheme photocatalytic mechanism demonstrated significant improvement in the photocatalytic enactment. The movement of the generated charges in the Z-scheme mechanism is slightly different compare to the type II heterojunction mechanism. In the type II mechanism, the excited electron from the *n-type* semiconductor will migrate to the conduction band of *p-type* semiconductor. While the produced holes from *p-type* semiconductor will be transferred to the valence band of *n-type* semiconductor [28]. However, the excited electrons at the conduction band of *p-type* semiconductor cannot migrate to the valence band of *p-type* semiconductor. Therefore, the photocatalytic reaction in this mechanism is focus on the low redox potential, which results in low photocatalytic activity.

In contrast, the excitation of electrons occurs at both junctions in the Z-scheme mechanism. The Z-scheme mechanism shows the movement of excited electrons from the valence band of *p-type* semiconductor to the conduction band. Then, the electrons at the conduction band of *p-type* semiconductor will migrate to *n-type* semiconductor and recombined with the holes at the valence band of *n-type* semiconductor. Unlike conventional p-n heterojunction, the Z-scheme mechanism is focusing the photocatalytic reaction at the bands with strong redox potentials [28]. Thus, resulting in high efficiency of photocatalytic abatement. Moreover, the suppression of charge recombination in the Z-scheme mechanism directed to the potential of Z-scheme heterojunction photocatalyst in wastewater purification. For instance, Chen and his co-workers have successfully fabricated the MoO₃/g-C₃N₄ composite photocatalyst which exhibited Z-scheme heterojunction mechanism [18]. In this study, the prepared catalyst is been study for the degradation of ofloxacin in the visible light irradiation. Impressively, about 94.4% of ofloxacin's degradation is observed in this study. In contrast, the removal of ofloxacin by commercial g-C₃N₄ only shows about 70% efficiency. This can be reasoned to the existence of the interaction between MoO₃ and g-C₃N₄ which significantly improved the photocatalytic properties. These findings infer that the development of Z-scheme heterojunction photocatalyst is very crucial in order to achieve high efficiency of antibiotic pollutants removal.

4. Conclusion and Future Prospects

Molybdenum oxide (MoO₃) has shown great potential as a photocatalyst material for wastewater remediation. The drawbacks of the MoO₃ photocatalyst have been successfully suppressed by some modification strategies. Interestingly, the modified MoO₃ based photocatalyst significantly enhanced the photocatalytic activity towards pharmaceutical pollutants abatement. The mechanistic of photocatalytic reaction for MoO₃ photocatalyst was also scrutinized comprehensively. The formation of Z-scheme heterojunction surprisingly improved the photocatalytic activity of the MoO₃ photocatalyst with the reduction in the recombination of excitons. These findings directed to the acknowledgment of photocatalysis as an efficient method for pharmaceutical pollutants removal and the potential of MoO₃ material in eliminating the pharmaceutical substrates in the contaminated waters.

The development of modified MoO₃ photocatalyst has provided substantial information for wastewater treatment. Nevertheless, some improvements can be applied to the MoO₃ photocatalyst before been tested for real wastewater remediation. In real wastewater treatment, the presence of foreign substances such as interference ions, organic compounds, and suspended particles cannot be neglected. Therefore, the modified MoO₃ photocatalyst should be first tested with real wastewater conditions. Besides, the immobilize of photocatalyst technique should be applied to avoid minimize the loss of photocatalyst during the process and the formation of suspended solids. Lastly, the total organic carbon (TOC) test should be carried out since the reduction in the intensity of parent targeted pharmaceutical pollutants does not represent the success of photocatalytic reaction as the formation of intermediate might poses more hazard potential to the human and environment.

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